Corrosion Inhibition Efficiency of Mild Steel in Hydrocloric Acid by Adding Theobroma Cacao Peel Extract

Yuli Yetri, Emriadi, Novesar Jamarun, and Gunawarman

Abstract---Inhibition and adsorption properties of *Theobroma cacao* peel polar extract (TCPE) addition on corrosion inhibition efficiency of 0.3%C mild steel in hydrochloric acid solution for various exposuring time, TCPE concentration and working temperature were investigated using weight loss test method. Electrochemical polarization test was also conducted to confirm the effectiveness of inhibition. Infrared spectrums of the samples were also evaluated to reveal compounds of the extract which controll the inhibition process. Morphology and local composition of sample surfaces were respectively examined by scanning electron microscope (SEM) and energy dispersive X-ray spectroscopy (EDX). Thermodynamic parameters such as energy activation, enthalpy, entropy and change in the free energy were then determined using related data. The result shows that the inhibition efficiency increases significantly up to 96.3% (by weight loss method) and 95.6% (by electrochemical polarization) with the increase of TCPE content. The optimum efficiency is obtained at TCPE concentration of 2,5% for exposuring time of 768h. However, the efficiency decreases slightly with increasing working temperature in the range of 303K-323K. The polarization curve shows the inhibitor behaves as a mixed inhibitor with the dominant cathodic inhibition. TCPE is identified to have functional groups of phenol, aromatic rings and ether. These compound is then adsorbed by the surface of the mild steel. The adsorption model is found to obey Langmuir adsorption isotherm. Surface condition is improved due to the adsorption and then formation of thin layer film protection in the surface of the steel. The addition of TCPE into HCl is effective to minimize corrosion attack on the mild steel.

Keywords----Corrosion, inhibitor, *Theobroma cacao* peel, Mild steel, Potensiodynamic

I. Introduction

CORROSION of mild steels is a problem in many industries that continuously attracted the attention of researchers. In some industrial processes such as cleaning with acid, etching, and pickling using aggressive media such as acids, bases, and salts, leads to corrode the steel. A number of ways such as material selection, cathodic or anodic protection, coatings and the use of corrosion inhibitors has been used to reduce corrosion of the installation industry[1-4]. Among them, corrosion inhibitors made of organic compounds has recently attracted to obtain eco-friendly inhibitors[2]. Some natural ingredients extracted from plants like henna leaf extract, green tea extract, mangrove wood tannins and flavonoid monomer has been introduced as new potential inhibitors[3,4]. However, such plants have already own ordinary applications that may hinder them for inhibitors. It is, therefore, agricultural wastes may be more reasonable to be used to make them more valueable.

Peels of cacao (*theobroma cacao*) plant is a large wasteful of cocoa production that is usually simply disposed and burried in the hole around cocoa plants. This kind of tropical plant are widely found in Indonesia espicially in hilly are of West Sumatera. Cocoa in the form of fresh fruit consists of 73% peel (and placenta 2% and 24.2% seed). Cocoa peels has found to gave enough tannin compounds[5]. This fact is allowing utilization of cacao peels as a natural inhibitor. This study is to determine the inhibitory power of cacao fruit peel extract by measuring corrosion rate and inhibition effeciency of the mild steel with addition of the exctract in acidic environment[6,7].

Yuli Yetri, Padang State Politechnic, and Department of Chemistry, Andalas University, Kampus Limau Manis Padang, Indonesia Emriadi, and Novesar Jamarun are with Department of Chemistry, Andalas University, Kampus Limau Manis Padang, Indonesia,

Gunawarman, Department of Mechanical Engineering, Andalas University Kampus Limau Manis Padang, Indonesia.

II. Research Methodology

Material and Procedures

Mild steel bar with a diameter of 12.0 mm was cut to obtain 2.0-4.0 mm thick of coin-like plates. The plate surfaces were then grinded by sand paper sizes of 400, 800, 1000, and 1500 as followed by polishing process using polish solution containing alumina powder. The smooth surface was then washed with detergent and rinsed with distilled water and acetone pa, and finally dried in an oven at 40^{o}C for 10 minutes. The samples were stored in a desiccator before corrosion process. The weight each samples was measured with digital balance prior to corrosion test. This weight is denoted as W_1.

Extraction-Process

Cocoa peels is cleaned of dirt, then chopped into small pieces and dried in the open air-dry for 14 days. Dry peels were then grinded to obtain powder dry. 1000 g cocoa peel powder was then put in 1 L maserator containing 70% methanol. The mixture was stirred in maserator for 4 days, and the result was subsequently filtered by using filter paper. The filtrate was put in a vacuum rotary evaporator at a temperature of 54-55 ^{0}C to obtain cacao concentrate. The result of the extract was put in brown bottles and qualitative analysis whether there is tannin or not. The crude extract of the cocoa peel extract was fractionated to obtain polar extract. The fractionation was conducted using hexane, and followed by using ethyl acetate. Finally, the remaining ethyl acetate fraction was obtained as polar fraction extract of cocoa peels. This polar extract was then used as a corrosion inhibitor.

Corrosion Test (Weight Loss).

Corrosion test was conducted by immersing the samples in the corrosive medium of 1.5 M hydrochloric acid (HCl) without and with addition of the extract for exposuring time of 48, 96, 192, 384 and 768 hours. The inhibition extract was put directly to the HCl solution with variations of the extract concentration of 0.5, 1.0, 1.5, 2.0 and 2.5%. After the corrosion process run for the specified time, the sample was lifted from the corrosion medium. It was then washed carefully with distilled water using a soft brush. Subsequently, it was rinsed with acetone, and then dried at room temperature. Finally, the weight of each sample after corrosion test (W_2) was measured by digital balance. The corrosion rate (V) of each sample was then determined by using following equation (equation 1).

$$V = (W_2 - W_1)/(A\ t) \qquad (1)$$

where:

W_1 = initial weight (prior to immersion)
W_2 = final weight (after immersion)
A = surface area of the sampel
t = exposure time

Furthermore, the inhibition efficiency (IE) of each sample was calculated by using equation 2 .

$$IE = (V_0 - V_1)/V_0 \qquad (2)$$

V_o = corrosion rate without inhibitor
V_1 = corrosion rate with the addition of inhibitor

Potentiodynamic Polarization Method

To determine the behavior of the active-passive corrosion test samples using potentiodynamic technique. Potentiodynamic polarization measurements performed in 1.5 M HCl medium, the variation of the concentration of inhibitor 0,0; 0.5; 1.0; 1.5; 2.0 and 2.5%. Electrodes are used, namely: Pt, Ag/AgCl, and mild steel. The third electrode is immersed in the cuvet containing the medium corrosive HCl without and with the inhibitor. Then, it was connected to and arranged potensiostat potential curves to obtaine the relationship between potential (E) vs. current (I).

Adsorption Isotherms

There are several types of adsorption isotherms that can be used as a reference in studying the adsorption mechanism of corrosion inhibitors, including the Langmuir, Freundlich and Temkin adsorption isotherm. Langmuir adsorption isotherm is the simplest model of adsorption, assuming that there is no interaction between molecules adsorbat[8]. A monolayer coating is formed, the maximum fraction of the closure; $\theta = 1$ (when the adsorbent surface saturated with adsorbate), homogeneous adsorbate surface so that each has a surface area equal to the bond energy, and the adsorbed molecules localized or not moving on surface.[9] The Langmuir isotherm expressed in equation 3 below:

$$\Theta/(1-\Theta) = KC \qquad (3)$$

where θ is the fraction of the surface covered by the inhibitor and C is the inhibitor concentration, and K is a constant. If the value of ΔG_{ads} negative or K greater than the higher adsorption energy. The results stated that the spontaneity of the adsorption process and the stability of the adsorbed layer on the surface adsorben.[10]

Surface Morphology

Surface morphology of the certain samples was examined by optical microscope and Hitachi S3400N Scanning Electron Microscopy. Analysis of elemental composition on the surface was also examined by SEM-EDX with EMAX software.

III. Results and Discussion

Corrosion Rate Analysis

Figure 1 shows the weight loss of steel in a variety of exposure time in 1.5 M HCl. The weight loss of the samples decreases with the increase of exposure time. This means that corrosion rate of the mild steel increases significantly with the increase of exposure time. On the other word, the increase of extract concentration decreases significantly the corrosion rate. This indicates that the increase number of extract on the steel surface is able to inhibit the corrosion. This is because of cocoa peel extract contains tannin compound. Tannin compound forms complex compounds with Fe (III) on the metal surface11. This is supported by FTIR test results which show some of peak is shifted that indicate interaction between the inhibitor with the steel surface. This complex compound will prevent attacks of corrosive ions on the metal surface, so that the corrosion rate will decrease. Thus, the polar extract of cocoa peels can be used as a natural inhibitor to inhibit the corrosion rate of steel.

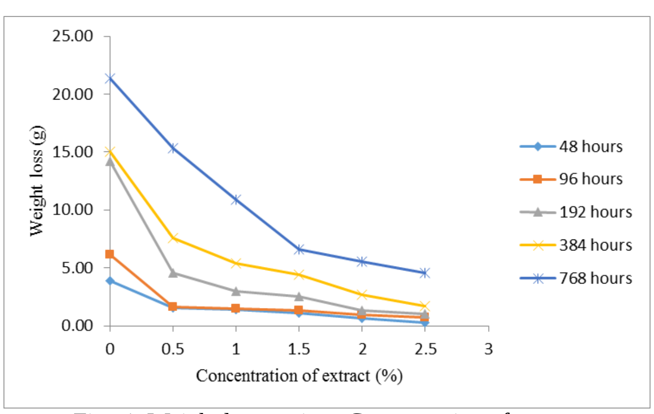


Fig. 1 Weight loss against Concentration of extract

Analysis of Potentiodynamic Polarization

Corrosion testing using the technique of polarization resistance is intended to observe at the sample resistance to oxidation when external potential is given. Resistance polarization is a fast method for determining the rate of corrosion without damaging metal and more accurate measurement results. The experimental result using polarization resistance is shown in Table 1.

TABLE 1
RESULT OF POTENTIODYNAMIC TESTING FOR HCL 1.5 M

No.	Inhibitor Conc. %V/V	Icorr mA cm^{-2}	Ecorr Vdec^{-1}	ba Vdec^{-1}	bc Vdec^{-1}	Rp Ωm^2	IE (Icorr)	IE (Rp)
1.	Blanko	0.0631	-0.28	2.40	1.71	6.8707	-	-
2.	0,5	0.0159	-0.275	2.80	2.30	34.4754	74.81	80.07
3.	1,0	0.0126	-0.25	3.67	2.57	52.0520	80.03	86.60
4.	1,5	0.0079	-0.20	2.00	1.60	57.3545	87.48	88.02
5.	2,0	0.0066	-0.22	5.25	2.63	115.2546	89.54	94.04
6.	2,5	0.0050	-0.28	5.71	2.67	157.9865	92.08	95.64

Table 1 shows the value of the free corrosion potential (Ecorr), corrosion current density (Icorr), polarization resistance (Rp) and corrosion rate of each metal in the different samples of each type of inhibitor concentrations is used. When the metal incorporated into the solution of the electrochemical reaction will occur at the interface between the metal and the solution. This reaction produces an electrochemical potential called the corrosion potential (Ecorr). This potential is determined by the amount of negative charge that is formed when the metal was introduced into the solution. The size of the potential price of corrosion samples indicate a tendency to undergo oxidation at while in corrosion media. If the free corrosion potential of the sample means that the sample is lower measurable easily oxidized, and otherwise. With the decrease of corrosion rate will increase the inhibition efficiency. Inhibition efficiency of the cocoa peel extract produced depends on the concentration of the inhibitor and corrosive media used. In the graph it can be seen that the efficiency of inhibition in hydrochloric acid corrosive media can reach 96.03% (weight loss), 95.64% (IE) and 92.08 (Icorr) inhibitor concentration of 2.5% as shown in Table 1. This is because in these conditions Fe-tannin complexes formed and covered the entire surface of the steel. Thus the surface coverage will also increase with the increase of inhibitor concentration, the relationship can be seen in Figure 2. Comparison of three methods for testing the efficiency of inhibition of polar extract of cocoa peels on mild steel shown in Figure 3.

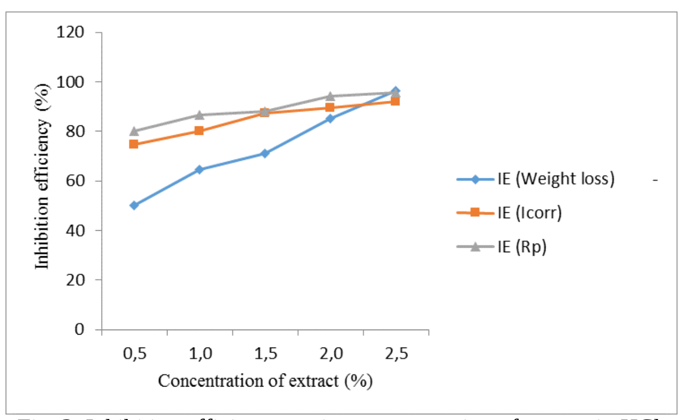


Fig. 3 Inhibition efficiency against concentration of extract in HCl 1,5 M

Thermodynamic parameters

Corrosion activation energy (Ea) of mild steel increases with the addition of the extract as shown in Table 2. But the change of enthalpy decreases with the addition of extract. The high value of Ea and ΔH causes the corrosion process is more difficult to occur. Thi is because of the metal will require greater energy to carry out the oxidation reaction. The decrease in Ea and ΔH values prove that the extract polar of cacao peels are involved on the reaction mechanism that occur on the surface of mild steel. This shows that the mechanism has chemical adsorption that is the transfer or sharing of inhibitors to the surface charge of mild steel[12].

FTIR Results

Figure 4 shows FTIR spectra of the cocoa peel extract and mild steel corrosion products after immersion in the solution with the addition of 2.5% extract. Figure 4a and 4b seen a significant difference because there are few dominant peaks disappear and new peaks appear while no significant change was in the same range as summarized in Table 3. Shift only identify the bond between the compound extract of the mild steel surface. FTIR results showed that the cocoa peel extract containing possible groups phenols , aromatic rings and ether groups.

TABLE II
THERMODYNAMIC PARAMETERS OF MILD STEEL ABSENCE AND PRESENCE OF POLAR EXTRACT OF CACAO (*THEOBROMA CACAO*) PEELS IN 1.5 M HCL.

No	Indikator	Ea(kJ/mol)	ΔH (kJ/mol)
1.	Blank	142.3782	217.6073
2.	Blank + 2.5% inhibitor	198.8048	196.1433

TABLE III
FTIR TRANSMITTANCE SPECTRA OF TCPE , CORROSION PRODUCT AND THEIR IDENTIFICATION

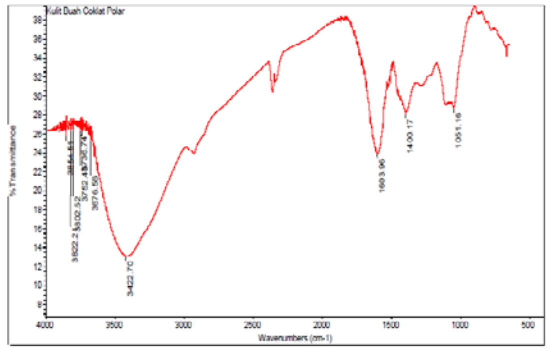
No	Peaks from FT-IR spectra, ν (cm^{-1})		Possible Groups
	TCPE	Corrosion Product	
1	-	620	Fe-H
2	-	835	Fe=O
3	1051	1019	C-O (ether)
4	1400	-	C-C=C(asimetric aromatic)
5	1603	1629	C=O
6	-	2363	H-C-H (phenol)
7	3422	3378	O-H (phenol)

There are several peaks in Figure 4a missing in Figure 4b, and some new peaks appear in Figure 4b. However, many peaks that appear in the same or adjacent frequencies. Identified functional groups of polar extract cacao peels (Figure 4a) are phenols, aromatic rings and ether. Most of these functional groups appear in the corrosion products but with little frequency shift. For example, CO functional groups that are at a frequency of 1051cm^{-1} shifted to 1019 cm^{-1}, C = O shift from 1603 cm^{-1} to 1629 cm^{-1}, whereas the OH shift from 3422 cm-1 to 3378 cm^{-1}. New peak appears at a frequency of 620 cm^{-1} is the Fe-H bond. Another new peak at 835 cm^{-1} is due to the strain allegedly Fe = O bond. These results indicate that there has been interaction and chemical bonding between metal compounds and extracts the surface area. Functional groups identified from existing peaks in both spectra is shown in full in Table 3

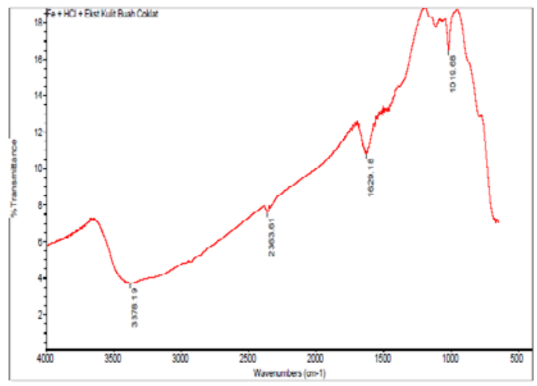
Effect of Temperature

The corrosion rate increases with the increase of temperature as can be seen in Table 4. Corrosion is the oxidation reaction. It means an increase in temperature will increase the rate of oxidation reaction which in this case is the rate of corrosion. Formation a coating layer on the surface of material will protect the surface of the steel so as to reduce the corrosion rate. It can be seen that with the efficiency of inhibition decreases with increasing temperature. This is due to the increased speed of oxidation of iron in the steel surface with increasing temperature, so the adsorbate from the peel extract cocoa will easily detached from the stell surface[13, 14].

Analysis of mild steel surface and the formation of a passive layer on the surface with the absence and presence of inhibitors was studied using Scanning Electron Microscopy with magnification of 2.0k are shown in Figure 6. Surface morphology of the initial specimens shows relatively smooth with small fine scratchs resulting from grinding and poleshing on the surface of mild steel (Fig. 6a). It can be also seen that the surface is flat, clean, non-porous and there are almost no holes. While, Figure 6b shows the surface morphology of the steel immersion in peels extracts of cocoa, which looks extract attached on the surface of mild steel.



(a)

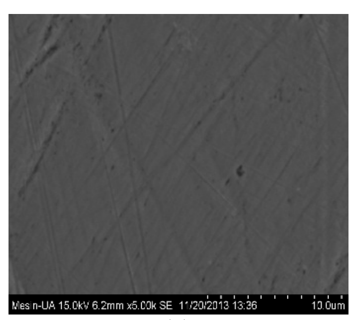


(b)

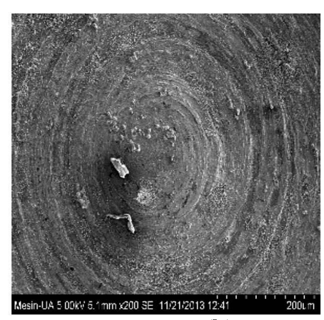
Fig. 4. FTIR spectra of a) TCPE b) adsorption layer formed on the mild steel surface after immersion in 1.5M HCl with 2.5 % TCPE for 8 days (196 h)

TABLE IV
EFFECT OF TEMPERATURE ON THE INHIBITION EFFICIENCY IS 6 HOURSIMMERSION IN 1.5 M HCL

Inhibitor Conc., %V/V	Inhibition Efficiency, %				
	303 K	308 K	313 K	318 K	324 K
0	-	-	-	-	-
0.5	35.99	30.28	27.84	18.99	7.23
1	43.92	41.54	36.77	31.62	17.85
1.5	57.19	53.25	48.78	46.98	36.75
2	69.39	64.97	60.91	58.09	47.16
2.5	83.91	77.78	69.92	64.56	58.05



(a)



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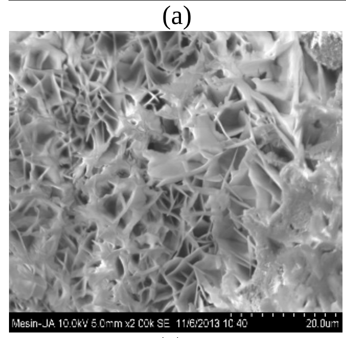

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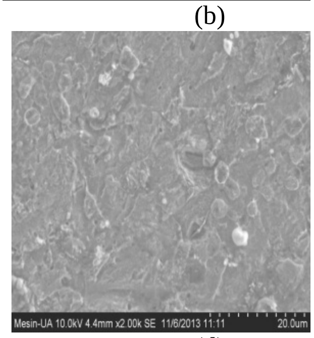

(d)

Fig. 6 SEM images of Mild steel in 1.5M HCl after 32 days immersion at room temperature (a) before immersion (polished), (b) immersion inhibitor, (c) immersion HCl without inhibitor, and (d) with 2.5 % inhibitor.

Surface of mild steel in HCl with the absence of polar extract cocoa peels (Figure 6c) shows corrosion products is formed. It is very clear that there is heavy damage on the surface. But the presence of cacao peel extract (Figure 6d) minimize corrosion products and any pits on the surface of the steel by forming a passive layer on the surface. This layer as a barrier

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against corrosive ions on the surface of mild steel so slow and electrochemical reactions corrosion rate will be eventually decreased.[16, 17]

SEM-EDX Analysis

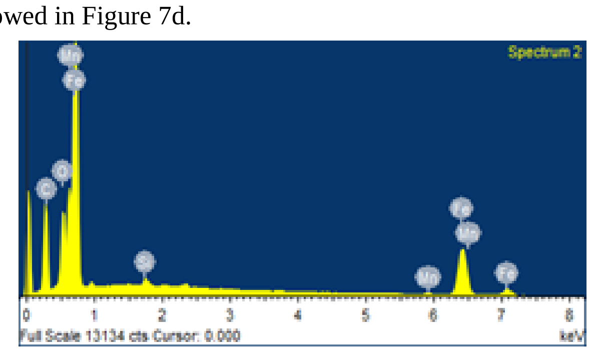
Elements of C and Fe on the surface of mild steel in 1.5 M HCl after immersion for 32 days with and without the extract can be seen in Figure 7 and Table 5. The atomic percentage of C increases significantly from 0.3% to 16.90%. This proves that the C atoms of the molecule of cocoa peel extract adsorbed on the steel surface to form a passive layer on the surface of mild steel.[17] While the percentage of elemental Fe atoms decreases in the presence of polar extract of cocoa peels, from 98.79% to 37.43%.

This suggests that the Fe to form organometallic complexes with polar extract cocoa peels so the percentage of detected Fe atoms into smaller[18]. The elements were detected in the initial O in Figure 7a does not exist, and in Figure 7b is detected with a low percentage.

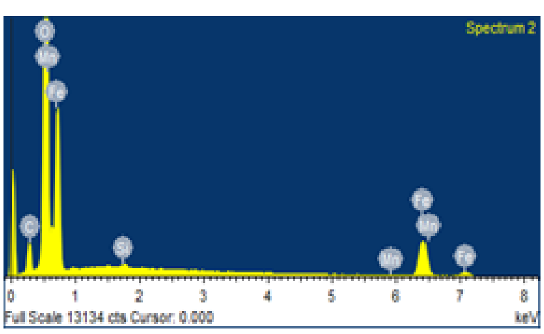
TABLE V
RECAPITULATION OF ELEMENTS AND OXIDES ARE IDENTIFIED ON SEM-EDX TESTING

No	Treatment	Content Elements (Wt %)		
		C	Fe	O
1.	Mild steel	0.32	91.43	-
2.	Mild steel + Extract	11.50	82.13	5.54
3.	Mild steel + HCl 1.5M	8.57	23.80	66.89
4.	Mild steel + HCl 1.5M + Extract	26.40	36.14	34.54

While, there is an increase of oxygen percentage to 63.54% for immersion in 1.5M HCl corrosive media without inhibitors. So, the oxide is formed quickly by an attack from the corrosive ions HCl. But with the contribution of inhibitors of the extract, the attacks of the corrosive ions can be prevented by forming a passive layer in the form of organometallic complexes on the surface of mild steel. Thus, the corrosion rate becomes slow and oxides formed as evidenced less than the percentage of such low levels of O showed in Figure 7d.



[a]



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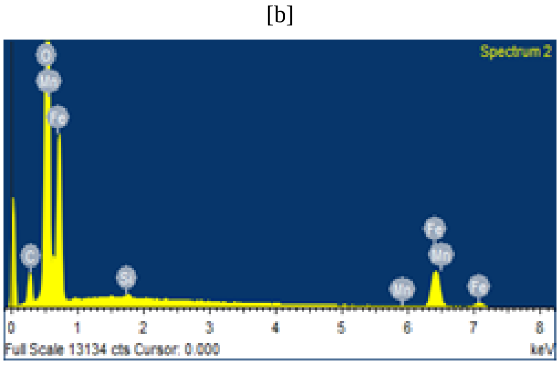

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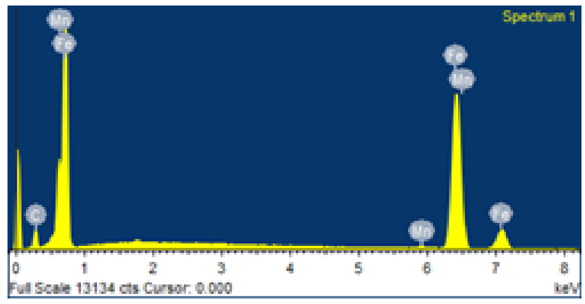

[d]

Fig. 7 SEM-EDX graph (a) Surface Mild steel, (b). Mild steel with extracts, (c). Mild Steel in HCl 1.5M, (d). Mild steel in HCl with 2.5% extract

IV. CONCLUSION

Corrosion behavior of mild steel in 1.5M HCl solution with and without addition of *theobroma cacao* peel polar extract (TCPE) for various exposuring time and working temperatures have been investigated. This study concludes that;

1. The corrosion rate of mild steel decreases significantly with the increase of TCPE concentration.
2. The inhibition efficiency (IE) of TCPE reach 96.3% by weight loss method and that are comparable with IE result of potentiodynamic polarization (92.1%) and polarization resistance method (95.6%).
3. Adsorption of polar extracts of cocoa peels in mild steel surface followed Langmuir adsorption isotherm with indication adsorpsi mechanism is chemical adsorption.
4. Potentiodynamic polarization analysis shows that the value of the corrosion current decreases from 0.0631 becomes 0.0050 mA/cm2, and the types of inhibitors are mixed type corrosion inhibitor with predominantly cathodic inhibitor.
5. Surface morphologies show the increase of a passive layer of protection area with the increase of TCPE concentration.

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